PROGRESS REPORT NO. II

A STUDY TO DEVELOP A MAGNESIUM OXIDE MATERIAL FOR STORAGE HEATERS

by

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ABSTRACT

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Ball-milled sintered magnesia of several different purity levels and calcined magnesia were pressed into right cylinders and fired to 1700°C (3092°F)/6 hours. Standard tests to determine the comparative mechanical properties of these materials were run and the results are summarized below. Development of a laboratory method for granulating magnesia and fabrication of various shapes is also briefly covered.

AUTHOR

STANDARD TESTS

High Temperature Creep

The loading and temperature variables used to start with were based on those to be encountered in the storage heater at Ames. Increased loading was not attempted in the 2200°C (3992°F) tests (this will be done in the near future), but arbitrary loading values in excess of maximum storage heater requirements were finally used in the 1700°C (3092°F) creep tests, because no measurable creep could be detected at the minimum load. Work is incomplete to date, and more meaningful results are anticipated when longer soak periods are used (maximum period used to date - 24 hours). Most of the work done so far has been of an exploratory nature, enabling us to learn which type and purity level of MgO exhibits the most promising creep resistance. Based on comparative tests, the 96% pure MgO does not possess adequate creep resistance, but the other types of MgO, 98% purity and above, appeared to be suitable.

Compressive Strength at Elevated Temperature

Some difficulties were encountered in choosing a ram material for the compressive strength equipment, but high purity alumina was finally used successfully. Tests run so far have been made at 1400°C (2552°F). Kaiser's calcined MgO was found to have the highest strength, approaching 16,000 PSI. The 96% pure sintered MgO exhibited the lowest, about 2300 PSI, suggesting that at this purity level, sufficient liquids are forming to weaken the structure of the test specimen.

Thermal Shock

Specimens were cycled in the region 1200°C (2192°F) to 2000°C (3632°F) at a heat/cool rate of 200°C (392°F) per minute. Kaiser's sintered K-Grain was the most resistant to shock under test conditions, and Kaiser's calcined MgO was the most susceptible.

Density

All specimens which underwent the standard tests were fired to 1700°C (3092 F) for 6 hours. Shrinkage due to further sintering was noted during creep tests at both 1700°C (3092°F) and 2200°C (3992°F). The sintered grain specimens appeared to be more prone to dimensional changes because of this shrinkage than specimens made from calcined MgO. Since structural stability is an important criterion for good storage heater media, sintering shrinkage must be reasonably complete at the contemplated operating temperatures. To date, sintered right cylinders have ranged in density from 76% to 96% of theoretical density, depending on the starting material and the forming techniques employed.

Vaporization

Although specific tests were not undertaken to determine MgO vaporization (see Progress Report No. I), two creep tests run at 2200°C (3992°F)/2 hours under similar conditions yielded some interesting secondary data. The 96% MgO specimen underwent a small weight loss, and the 99% MgO a considerable weight gain. From previous work at 2200°C (3992°F), we have been aware of the fact that vaporized MgO produced from the furnace lining tends to condense on the specimens as well as elsewhere. This phenomenon leads us to believe that, assuming a relatively constant rate of MgO condensation, the 96% MgO specimen has itself lost mass due to volatilization of low melting point constituents.

Granulation

We have been primarily concerned with the development of a laboratory method for granulating MgO, with emphasis on calcined MgO in a non-aqueous system. After testing and evaluating several resins and other organic materials, a satisfactory system was developed. Although not meant to produce more than modest amounts of granulated powder, it nevertheless will suit our needs at present.

FABRICATION OF SHAPES

Modified Spheres

Both Kaiser's sintered K-Grain (98% MgO) and Kaiser's calcined MgO were pressed into spheres and fired to 1700°C (3092°F). The mechanical behavior of the calcined MgO during the pressing operation tended to set up stresses and cracks in the specimen. Die function may be contributing to this problem.

Hexagonal Cored Shapes

Specimens were successfully pressed from Kaiser's sintered K-Grain in initial work to check on the proper functioning of the die parts. These specimens were fired to 1700°C (3092°F) for one hour. Maximum density observed was 76.7% of theoretical, and no visible cracks were present.

Isostatically-pressed Tubes

Our involvement with the standard test procedures and granulation study prevented the fabrication of additional tubes by isostatic pressing. This will be accomplished in the immediate future.

FUTURE WORK

Emphasis will be placed on relating microstructure to firing time/temperature, density, compressive strength and thermal shock resistance. Continued efforts will be made to fabricate tubes, modified spheres and cored brick with good structure and density.